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(54) Abstract Title
Coloured detergent particles

(57) A particulate laundry detergent composition comprises a major proportion of white or light-coloured particles, and a minor proportion of visually contrasting particles of material capable of imparting a colour to the resulting solution when the composition is dissolved in water. The visually contrasting particles may contain a fluorescent material, yielding a fluorescent wash liquor, or may contain a pH indicator which gives a coloured solution at high pH but a colourless or differently coloured solution at lower pH values. The visually contrasting particles and the visual effects in the wash liquor provide cues to the consumer, for example, to demonstrate the presence of a beneficial ingredient, or to indicate that more product should be added.

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DETERGENT COMPOSITIONSTECHNICAL FIELD

5

The present invention relates to laundry detergent powders, for use in laundering fabrics by hand or machine.

BACKGROUND AND PRIOR ART

10

It is well known to include visually contrasting particles, for example, coloured speckles or noodles, in laundry detergent powders. These may be included as a cue to the consumer, to indicate the presence of some specific ingredient, for example, bleach, or may simply be present to give the product an attractive appearance. However, the presence of the coloured speckles does not normally manifest itself in the wash liquor once the powder has been dissolved.

20

The present inventors now propose that the distinctive appearance conferred by coloured speckles or other visually contrasting particles may be carried over into the wash liquor itself. This may act as a further cue to the consumer, for example, to provide reassurance that some functional ingredient is present and effective in the wash liquor, or as a prompt to add more product where an insufficient amount has been used.

30

DEFINITION OF THE INVENTION

The present invention accordingly provides a particulate laundry detergent composition comprising a major proportion
5 of white or light-coloured particles and a minor proportion of visually contrasting particles of material capable of imparting a colour to the resulting solution when the composition is dissolved in water.

10

DETAILED DESCRIPTION OF THE INVENTIONThe visually contrasting particles

15

According to the invention, the visually contrasting particles are capable of conferring colour on the wash liquor when the product is dissolved.

20 The concentrations at which laundry powders are used will vary depending on the formulation and on the laundry method used, for example, handwash or machine wash. However, generally the visually contrasting particles should contain sufficient coloured material, and be present in a sufficient
25 amount, that a coloured solution is obtained at product concentrations of from 1 to 10 g/litre.

According to a first preferred embodiment of the invention, the visually contrasting particles comprise a fluorescent
30 material, whereby a fluorescent wash liquor may be obtained.

Examples of suitable materials include fluorescein (orange/yellow particles, fluorescent yellow solution) and rhodamine B (deep purplish pink particles, fluorescent pink solution).

5

The fluorescent materials may be deposited onto particulate carrier materials, for example, sodium metasilicate or sodium citrate, to prepare particles suitable for incorporation into laundry detergent powders.

10

As well as providing an unusual and attractive appearance in both the dry product and the wash liquor, the fluorescent materials may deliver some fluorescer to the fabrics which makes white and light-coloured fabrics appear more white and
15 bright.

According to a second preferred embodiment of the invention, aimed especially at the handwash, the visually contrasting particles comprise a pH indicator which is coloured at high
20 pH and colourless, or differently coloured, at lower pH values. In this embodiment of the invention the wash liquor will initially be coloured, and as soiled fabrics are added and the pH falls, the liquor will become colourless, or change colour. This acts as a cue to the consumer that
25 the product has been exhausted, or that insufficient product has been used, and that more should be added.

In this embodiment of the invention, it is necessary to select an indicator that changes colour at an appropriate
30 pH. This will generally be in the region of 7 to 10.

Preferably the colour change occurs at a pH within the range of from 8 to 10.

It is also preferred to use an indicator which initially, at
5 higher pH values, provides a blue solution. Some blue colour is then delivered to the fabrics, which for white fabrics improves whiteness and brightness and masks any yellowing that may have occurred in previous washes.

10 A suitable indicator is thymolphthalein which provides a blue solution at higher pH values and is colourless at lower pH values. The colour change occurs at a pH of about 9.

As with the fluorescent materials, indicators may be
15 deposited onto particulate carrier materials, for example, sodium metasilicate or sodium citrate, to prepare particles suitable for incorporation into laundry detergent powders.

The particles containing fluorescer or indicator may
20 suitably be present in the laundry detergent powders in amounts of from 0.2 to 5 wt%, preferably from 0.5 to 2 wt%, more preferably about 1 wt%.

They are most suitably incorporated in particulate laundry
25 detergent compositions by dry mixing.

Detergent compositions

The laundry detergent compositions of the invention also contain other conventional detergent ingredients. Essential
5 ingredients are surfactants (detergent-active compounds) and detergency builders, and other conventional ingredients may optionally be present.

Detergent compositions of the invention may suitably
10 comprise from 5 to 60 wt% of one or more detergent surfactants and from 10 to 80 wt% of one or more detergency builders, and optionally other detergent ingredients to 100 wt%.

15 The detergent compositions will contain, as essential ingredients, one or more detergent active compounds (surfactants) which may be chosen from soap and non-soap anionic, cationic, nonionic, amphoteric and zwitterionic detergent active compounds, and mixtures thereof.

20 Many suitable detergent active compounds are available and are fully described in the literature, for example, in "Surface-Active Agents and Detergents", Volumes I and II, by Schwartz, Perry and Berch.

25 The preferred detergent active compounds that can be used are soaps and synthetic non-soap anionic and nonionic compounds.

Anionic surfactants are well-known to those skilled in the
30 art. Examples include alkylbenzene sulphonates, particularly linear alkylbenzene sulphonates having an alkyl

chain length of C₈-C₁₅; primary and secondary alkylsulphates, particularly C₈-C₁₅ primary alkyl sulphates; alkyl ether sulphates; olefin sulphonates; alkyl xylene sulphonates; dialkyl sulphosuccinates; and fatty acid ester sulphonates.

- 5 Sodium salts are generally preferred.

Nonionic surfactants that may be used include the primary and secondary alcohol ethoxylates, especially the C₈-C₂₀ aliphatic alcohols ethoxylated with an average of from 1 to
10 20 moles of ethylene oxide per mole of alcohol, and more especially the C₁₀-C₁₅ primary and secondary aliphatic alcohols ethoxylated with an average of from 1 to 10 moles of ethylene oxide per mole of alcohol. Non-ethoxylated nonionic surfactants include alkylpolyglycosides, glycerol
15 monoethers, and polyhydroxyamides (glucamide).

Cationic surfactants that may be used include quaternary ammonium salts of the general formula R₁R₂R₃R₄N⁺ X⁻ wherein the R groups are long or short hydrocarbyl chains, typically
20 alkyl, hydroxyalkyl or ethoxylated alkyl groups, and X is a solubilising cation (for example, compounds in which R₁ is a C₈-C₂₂ alkyl group, preferably a C₈-C₁₀ or C₁₂-C₁₄ alkyl group, R₂ is a methyl group, and R₃ and R₄, which may be the same or different, are methyl or hydroxyethyl groups); and cationic
25 esters (for example, choline esters).

In an especially preferred cationic surfactant of the general formula R₁R₂R₃R₄N⁺ X⁻, R₁ represents a C₈-C₁₀ or C₁₂-C₁₄ alkyl group, R₂ and R₃ represent methyl groups, and R₄
30 presents a hydroxyethyl group.

Amphoteric surfactants, for example, amine oxides, and zwitterionic surfactants, for example, betaines, may also be present.

5 Preferably, the quantity of anionic surfactant is in the range of from 5 to 50% by weight of the total composition. More preferably, the quantity of anionic surfactant is in the range of from 8 to 35% by weight.

10 Nonionic surfactant, if present, is preferably used in an amount within the range of from 1 to 20% by weight.

The total amount of surfactant present is preferably within the range of from 5 to 60 wt%.

15

The compositions may suitably contain from 10 to 80%, preferably from 15 to 70% by weight, of detergency builder. Preferably, the quantity of builder is in the range of from 15 to 50% by weight.

20

The detergent compositions may contain as builder a crystalline aluminosilicate, preferably an alkali metal aluminosilicate, more preferably a sodium aluminosilicate (zeolite).

25

The zeolite used as a builder may be the commercially available zeolite A (zeolite 4A) now widely used in laundry detergent powders. Alternatively, the zeolite may be maximum aluminium zeolite P (zeolite MAP) as described and claimed

30 in EP 384 070B (Unilever), and commercially available as Doucil (Trade Mark) A24 from Crosfield Chemicals Ltd, UK.

Zeolite MAP is defined as an alkali metal aluminosilicate of zeolite P type having a silicon to aluminium ratio not exceeding 1.33, preferably within the range of from 0.90 to 1.33, preferably within the range of from 0.90 to 1.20.

5

Especially preferred is zeolite MAP having a silicon to aluminium ratio not exceeding 1.07, more preferably about 1.00. The particle size of the zeolite is not critical. Zeolite A or zeolite MAP of any suitable particle size may
10 be used.

Also preferred according to the present invention are phosphate builders, especially sodium tripolyphosphate. This may be used in combination with sodium orthophosphate,
15 and/or sodium pyrophosphate.

Other inorganic builders that may be present additionally or alternatively include sodium carbonate, layered silicate, amorphous aluminosilicates.

20

Organic builders that may be present include polycarboxylate polymers such as polyacrylates and acrylic/maleic copolymers; polyaspartates; monomeric polycarboxylates such as citrates, gluconates, oxydisuccinates, glycerol mono-di-
25 and trisuccinates, carboxymethyloxysuccinates, carboxy-methyloxymalonates, dipicolinates, hydroxyethyliminodiacetates, alkyl- and alkenylmalonates and succinates; and sulphonated fatty acid salts.

30 Organic builders may be used in minor amounts as supplements to inorganic builders such as phosphates and zeolites.

Especially preferred supplementary organic builders are citrates, suitably used in amounts of from 5 to 30 wt %, preferably from 10 to 25 wt %; and acrylic polymers, more especially acrylic/maleic copolymers, suitably used in
5 amounts of from 0.5 to 15 wt %, preferably from 1 to 10 wt%.

Builders, both inorganic and organic, are preferably present in alkali metal salt, especially sodium salt, form. Detergent compositions according to the invention may also
10 suitably contain a bleach system, although non-bleaching formulations are also within the scope of the invention.

The bleach system is preferably based on peroxy bleach compounds, for example, inorganic persalts or organic
15 peroxyacids, capable of yielding hydrogen peroxide in aqueous solution. Suitable peroxy bleach compounds include organic peroxides such as urea peroxide, and inorganic persalts such as the alkali metal perborates, percarbonates, perphosphates, persilicates and persulphates. Preferred
20 inorganic persalts are sodium perborate monohydrate and tetrahydrate, and sodium percarbonate. The peroxy bleach compound is suitably present in an amount of from 5 to 35 wt%, preferably from 10 to 25 wt%.

25 The peroxy bleach compound may be used in conjunction with a bleach activator (bleach precursor) to improve bleaching action at low wash temperatures. The bleach precursor is suitably present in an amount of from 1 to 8 wt%, preferably from 2 to 5 wt%.

Preferred bleach precursors are peroxycarboxylic acid precursors, more especially peracetic acid precursors and peroxybenzoic acid precursors; and peroxycarbonic acid precursors. An especially preferred bleach precursor
5 suitable for use in the present invention is N,N,N',N'-tetracetyl ethylenediamine (TAED).

A bleach stabiliser (heavy metal sequestrant) may also be present. Suitable bleach stabilisers include
10 ethylenediamine tetraacetate (EDTA) and the polyphosphonates such as Dequest (Trade Mark), EDTMP.

The detergent compositions may also contain one or more enzymes. Suitable enzymes include the proteases, amylases
15 cellulases, oxidases, peroxidases and lipases usable for incorporation in detergent compositions.

Preferred proteolytic enzymes (proteases) are catalytically active protein materials which degrade or alter protein types
20 of stains when present as in fabric stains in a hydrolysis reaction. They may be of any suitable origin, such as vegetable, animal, bacterial or yeast origin.

Proteolytic enzymes or proteases of various qualities and origins and having activity in various pH ranges of from 4-12
25 are available. Proteases of both high and low isoelectric point are suitable.

Other enzymes that may suitably be present include lipases, amylases, and cellulases including high-activity cellulases
30 such as Carezyme ex Novo.

In particulate detergent compositions, detergency enzymes are commonly employed in granular form in amounts of from about 0.1 to about 3.0 wt%. However, any suitable physical form of enzyme may be used in any effective amount.

5

Antiredeposition agents, for example, cellulose esters and ethers, for example sodium carboxymethyl cellulose, may also be present.

- 10 The compositions may also contain soil release polymers, for example sulphonated and unsulphonated PET/POET polymers, both end-capped and non-end-capped, and polyethylene glycol/polyvinyl alcohol graft copolymers such as Sokolan (Trade Mark) HP22.

15

Especially preferred soil release polymers are the sulphonated non-end-capped polyesters described and claimed in WO 95 32997A (Rhodia Chimie).

- 20 Other ingredients that may be present include solvents, hydrotropes, fluorescers, photobleaches, foam boosters or foam controllers (antifoams) as appropriate, sodium carbonate, sodium bicarbonate, sodium silicate, sodium sulphate, calcium chloride, other inorganic salts, flow aids
25 such as silicas and amorphous aluminosilicates, fabric conditioning compounds, and perfumes.

- Powders of low to moderate bulk density may be prepared by spray-drying a slurry, and optionally postdosing (dry-
30 mixing) further ingredients. "Concentrated" or "compact" powders may be prepared by mixing and granulating processes,

for example, using a high-speed mixer/granulator, or other non-tower processes. In both types of powder, as previously indicated, the visually contrasting particles are preferably incorporated by postdosing (dry mixing).

5

EXAMPLES

The invention will now be illustrated in further detail by means of the following Examples, in which parts and percentages are by weight unless otherwise stated.

EXAMPLES 1 to 3

15 Preparation of visually contrasting particles

Example 1: Fluorescein crystals

20 Fluorescein (ex Merck) was dissolved in ethanol and the solution sprayed onto anhydrous sodium metasilicate (ex Ausimont), and the ethanol was then evaporated off. Yellow/orange crystals were obtained.

25 Example 2: Rhodamine B crystals

Rhodamine B (ex Merck) was dissolved in ethanol and the solution sprayed onto sodium citrate (ex Haarman & Reimer). The ethanol was evaporated off to leave purplish pink
30 crystals.

Example 3: Thymolphthalein crystals

The blue dye thymolphthalein was dissolved in a mixture of 70 wt% ethanol and 30 wt% water (the use of water intensifies the blue colour). The solution was sprayed onto anhydrous sodium metasilicate particles (ex Ausimont). The ethanol and water were evaporated off to give deep blue crystals.

10

EXAMPLE 4Detergent powder containing fluorescent crystals

A detergent powder having the following formulation was prepared by conventional spray-drying and postdosing techniques. The fluorescent particles were postdosed.

Ingredient	weight %
Sodium linear alkylbenzene sulphonate	36.72
Sodium tripolyphosphate	22.18
Sodium alkaline silicate	10.71
Sodium sulphate	15.45
Acrylate/maleate copolymer (Sokalan CP5 ex BASF)	3.06
Sodium carboxymethyl cellulose	0.57
Fluorescer	0.81
Fluorescein crystals of Example 1	1.00
Water and minor impurities to	100.00

The composition consisted of a major proportion of white particles with a minor but very noticeable proportion of bright orange/yellow particles.

20

A sample of the composition was dissolved in water to a concentration of 4 g/litre. The solution was fluorescent yellow in colour.

5 EXAMPLE 5

Detergent powder containing fluorescent crystals

Example 4 was repeated using the rhodamine B crystals of Example 2 (1 wt%) instead of the fluorescein crystals of
10 Example 1. The composition consisted of a major proportion of white particles with a minor but very noticeable proportion of vividly coloured purplish pink particles. A sample of the composition was dissolved in water to a concentration of 4 g/litre. The solution was fluorescent
15 pink in colour.

EXAMPLE 6

Detergent composition containing indicator particles

20 The procedure of Example 4 was repeated using the thymolphthalein crystals of Example 3 (1 wt%) instead of the fluorescein particles. The composition consisted of a major proportion of white particles with a minor but very noticeable proportion of dark blue particles.
25 A sample of the composition was dissolved in water to a concentration of 4 g/litre. The solution was blue in colour. The pH was about 11.

The pH was lowered to 9 by addition of dilute aqueous acid.
30 The solution became colourless.

CLAIMS

- 1 A particulate laundry detergent composition comprising
5 a major proportion of white or light-coloured particles and
a minor proportion of visually contrasting particles of
material capable of imparting a colour to the resulting
solution when the composition is dissolved in water.
- 10 2 A composition as claimed in claim 1, which provides a
coloured solution when dissolved at a concentration within
the range of from 1 to 10 g/l.
- 15 3 A composition as claimed in claim 1 or claim 2, wherein
the visually contrasting particles are present in an amount
of from 0.2 to 5 wt%, preferably from 0.5 to 2 wt%.
- 20 4 A composition as claimed in any preceding claim,
wherein the visually contrasting particles comprise a
fluorescent material.
- 25 5 A composition as claimed in claim 4, wherein the
particles comprise fluorescein or rhodamine B.

6 A composition as claimed in any one of claims 1 to 3, wherein the visually contrasting particles comprise an indicator which is coloured at higher pH values and colourless or differently coloured at lower pH values.

5

7 A composition as claimed in claim 6, wherein the indicator has a colour change at a pH within the range of from 8 to 10.

10

8 A composition as claimed in claim 6 or claim 7, wherein the visually contrasting particles comprise thymolphthalein.

15

9 A composition as claimed in any preceding claim, which comprises from 5 to 60 wt% of detergent-active material, from 10 to 80 wt% of detergency builder, from 0.5 to 5 wt% of visually contrasting particles, and optionally other
20 detergent ingredients to 100 wt%.

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Application No: GB 0001546.1
Claims searched: 1-9

Examiner: J. P. Bellia
Date of search: 2 May 2000

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Databases searched:

UK Patent Office collections, including GB, EP, WO & US patent specifications, in:

UK Cl (Ed.R): C5D (DDX, DHC, DJA)

Int Cl (Ed.7): C11D3/40, C11D17/04

Other: ONLINE: EPODOC, WPI, JAPIO

Documents considered to be relevant:

Category	Identity of document and relevant passage	Relevant to claims
A	GB 0 801 018 (THOMAS HEDLEY & CO) See whole document	-
X	WO 99/07817 A1 (PROCTER & GAMBLE) See page 3 line 1-14; page 4 line 15-31 & page 12 line 24-27	1-3
X	WPI Abstract Accession No. 1975-24818W [15] & JP 511016048 B (DAINICHISEIKA COL CHEMS) See abstract	1-9

X	Document indicating lack of novelty or inventive step	A	Document indicating technological background and/or state of the art
Y	Document indicating lack of inventive step if combined with one or more other documents of same category.	P	Document published on or after the declared priority date but before the filing date of this invention.
&	Member of the same patent family	E	Patent document published on or after, but with priority date earlier than, the filing date of this application.